



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Ginosar et al.

Serial No.: 09/554,708

Filed: July 31, 2000

For: PROCESS FOR PRODUCING  
BIODIESEL LUBRICANTS, AND FUEL  
AND LUBRICANT ADDITIVES IN A  
CRITICAL FLUID MEDIUM

Confirmation No.: 5703

Examiner: M. Medley

Group Art Unit: 1714

Attorney Docket No.: LIT-PI-099

CERTIFICATE OF MAILING

I hereby certify that this correspondence along with any attachments referred to or identified as being attached or enclosed is being deposited with the United States Postal Service as First Class Mail on the date of deposit shown below with sufficient postage and in an envelope addressed to the Commissioner for Patents, Washington, D.C. 20231.

3/3/2004  
Date

Mandy Landon  
Signature

Mandy Landon  
Name (Type/Print)

AFFIDAVIT

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Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

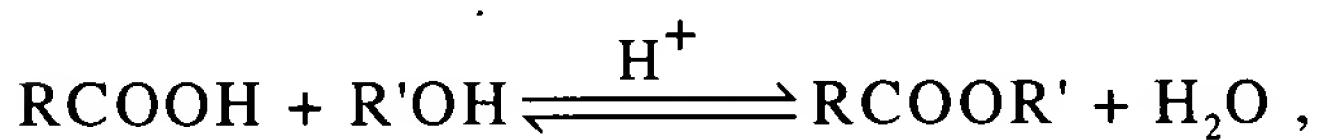
Sir:

I, Robert V. Fox, being duly sworn, depose and say that:

- (1) I, Robert V. Fox, a co-inventor of the above-referenced patent application, am a citizen of the United States of America currently residing at 3688 Woodhaven Lane, Idaho Falls, Idaho 83404. I received a Ph.D. in Chemistry from the University of Idaho in 2003. I have been employed at the Idaho National Engineering and Environmental Laboratory ("INEEL") since 1989. The INEEL is operated for the Department of Energy by Bechtel

BWXT Idaho, LLC (and its predecessor entities), the assignee of record of the above-referenced patent application.

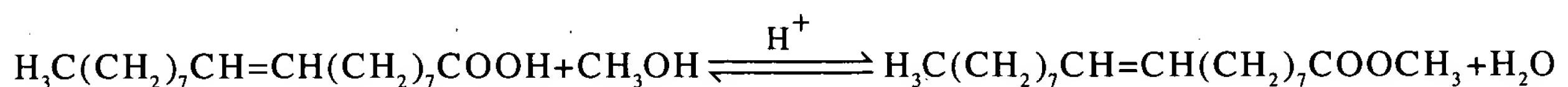
- (2) Much of our research and development activity at the INEEL is directed toward producing biofuels as alternatives to petroleum-based fuels by reacting fats and oils, such as triglycerides and free fatty acids, to produce alkyl esters of triglycerides.
- (3) It is well understood in the art that glycerol has a chemical formula of C<sub>3</sub>H<sub>8</sub>O<sub>3</sub> and a structure of CH<sub>2</sub>OHCHOHCH<sub>2</sub>OH.
- (4) Vieville *et al.*, "Esterification of Oleic Acid by Methanol Catalyzed by p-Toluenesulfonic Acid and the Cation-Exchange Resins K2411 and K1481 in Supercritical Carbon Dioxide, Industrial Engineering Chemical Research, Vol. 32, No. 9, pp. 2065-2068, September 1993 ("Vieville"), Exhibit A hereto, discloses a method of esterifying oleic acid by methanol in supercritical carbon dioxide ("CO<sub>2</sub>"). Vieville discloses using oleic acid as a starting material, which is well understood in the art to be a fatty acid or carboxylic acid. It is well understood in the art that oleic acid has a chemical formula of C<sub>18</sub>H<sub>34</sub>O<sub>2</sub> and a structure of H<sub>3</sub>C(CH<sub>2</sub>)<sub>7</sub>CH=CH(CH<sub>2</sub>)<sub>7</sub>COOH.
- (5) It is well understood in the art that the esterification of a fatty acid proceeds by the following reaction:



as described in Advanced Organic Chemistry, 4<sup>th</sup> Ed, p. 393-396, John Wiley and Sons Inc. (1992), Exhibit B hereto. The reaction is accomplished in the presence of an acid (H<sup>+</sup>) if there is an excess of alcohol to drive the reaction equilibrium to the right.

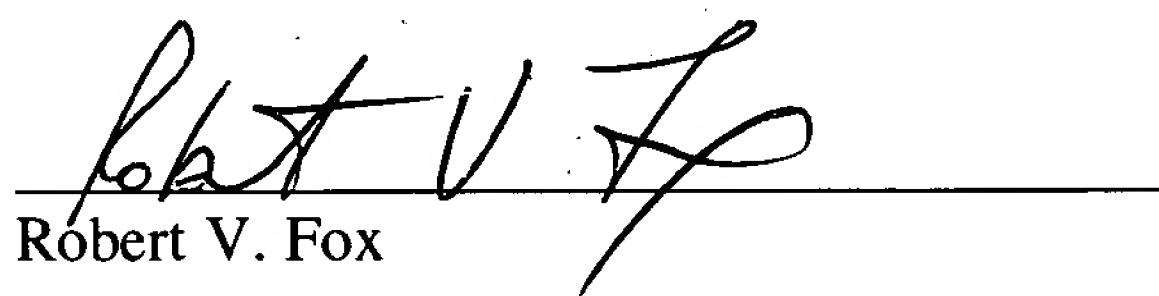
- (6) In the esterification reaction in Vieville, R in the above reaction is H<sub>3</sub>C(CH<sub>2</sub>)<sub>7</sub>CH=CH(CH<sub>2</sub>)<sub>7</sub>, R' is methanol, and H<sup>+</sup> is p-toluenesulfonic acid. The reaction

of oleic acid and methanol in the presence of p-toluenesulfonic acid produces methyl oleate and water. Therefore, the esterification reaction in Vieville is the following reaction:



- (7) As shown in paragraph 6, methyl oleate and water are produced by the esterification reaction of Vieville. Glycerol ( $\text{CH}_2\text{OHCHOHCH}_2\text{OH}$ ) is not produced in the reaction.
- (8) It is also well understood in the art that glycerol is produced when one of the starting materials of the reaction is a triglyceride.
- (9) The esterification reaction described in Vieville does not include any triglycerides as starting materials. Therefore, glycerol is not produced in the reaction of Vieville.

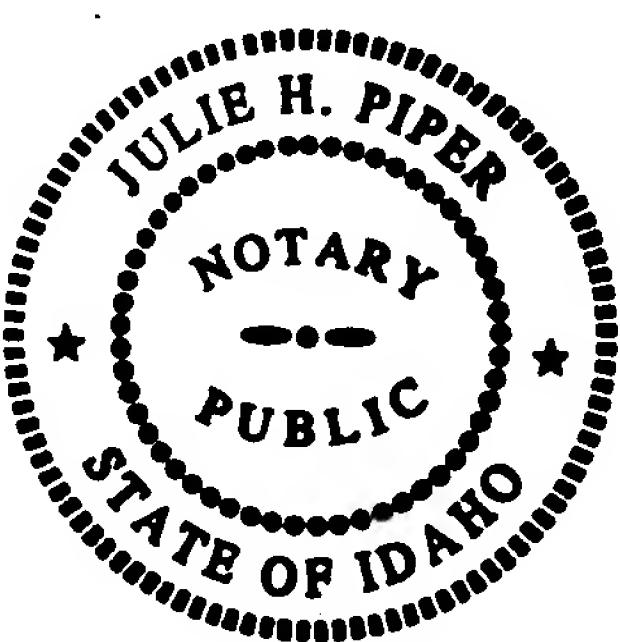
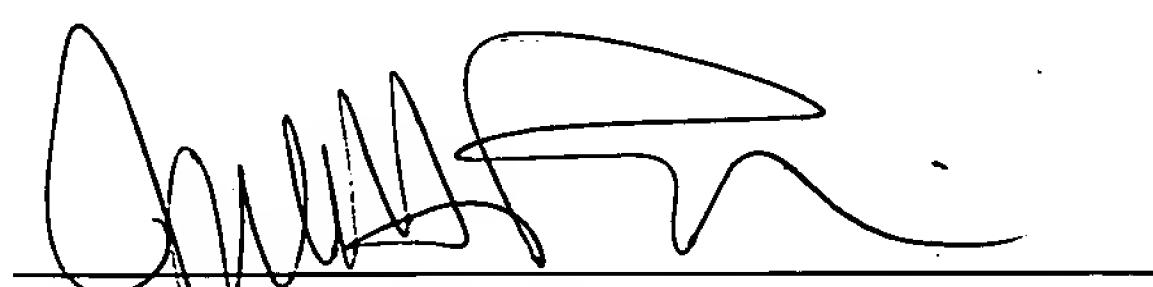
Further deponent sayeth not.



Robert V. Fox

STATE OF Idaho )  
ss.  
COUNTY OF Bonneville

BEFORE ME, the undersigned authority, on this 2nd day of MARCH, 2004, personally appeared Robert V. Fox, personally known or proven by satisfactory documentary evidence to me to be the person whose name is subscribed to the foregoing instrument and that he swore and subscribed to the same of his own free will.

Notary or Consular Officer  
Julie H. Piper

Commission Expires 8/18/09